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(54) Title: PROCESS FOR PREPARING HIGH BULK DENSITY DETERGENT COMPOSITIONS

(57) Abstract

A process for preparation of a granular detergent composition which process comprises the steps of (i) producing a pumpable feedstock comprising an acid precursor of an anionic surfactant; (ii) partially neutralising the acid precursor in the feedstock; (iii) feeding the feedstock into a high-speed mixer/densifier and completely neutralising the partially neutralised acid precursor therein, to form a granular detergent material; and (iv) drying and/or cooling the product of step (ii).

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PROCESS FOR PREPARING HIGH BULK DENSITY DETERGENT COMPOSITIONS

FIELD OF THE INVENTION

The present invention relates to a process for preparing a granular detergent composition or component having a high bulk density and good powder properties. More in particular, it relates to a process for the continuous preparation of such detergent compositions. Furthermore, it relates to a granular detergent composition obtainable by the process of the present invention.

BACKGROUND OF THE INVENTION

Recently there has been considerable interest within the detergents industry in the production of detergent powders having a relatively high bulk density, for example 550 g/l and above.

Generally speaking, there are two main types of processes by which detergent powders can be prepared. The first type of process involves spray-drying and aqueous detergent slurry in a spray-drying tower. In the second type of process the various components are dry-mixed and optionally agglomerated with liquids, e.g. nonionics. The latter kind of process is more suited to the production of powders having a relatively high bulk density. That is primarily because the chemical composition of the slurry used in the spray drying process markedly affects the bulk density of the granular product. This bulk density can only be significantly increased by increasing the content of relatively dense sodium sulphate. However, high levels of sodium sulphate do not contribute to detergency, so that the overall performance of the powder in the wash is thereby reduced.

One dry-mix process suitable for production of relatively high density products is described in European Patent Specification EP-A-0 420 317. This involves reacting a liquid acid precursor of an anionic surfactant with an alkaline inorganic material in a high-speed mixer/densifier, treating the material in a moderate-speed granulator/densifier, and finally drying and/or cooling the material. The heat of the neutralization reaction between the acid surfactant precursor and the alkaline material is used to bring the starting material into a deformable state, and results in densification of the detergent composition.

When the anionic surfactant should be formed in the high-speed mixer/agglomerator by neutralisation of the corresponding acid in a very dry powder having a high moisture absorbing capacity, a strong alkaline liquid needs to be added to initiate the neutralisation reaction. In such a case a problem has been found to arise, whereby dark yellow spots are formed in the final product when the perfume is sprayed on. Not limited by any theory, it is believed that this is caused by reaction of alkaline spots in the powder and the perfume.

SUMMARY OF THE INVENTION

This problem has been circumvented by the present invention by partially neutralising the acid precursor prior to dosing and final neutralisation in the high-speed mixer/densifier.

Thus, one aspect of the invention provides a process for the preparation of a granular detergent composition which process comprises the steps of

(i) producing a pumpable feedstock comprising an acid precursor of an anionic surfactant;

(ii) partially neutralising the acid precursor in the feedstock;

(iii) feeding the feedstock into a high-speed mixer/densifier and further neutralising any partially neutralised acid precursor therein, to form a granular detergent material; and

(iv) drying and/or cooling the resultant granular detergent material.

In a second aspect, the invention provides a granular detergent composition or component prepared by this process.

DETAILED DESCRIPTION OF THE INVENTION

The composition so prepared may include perfume, preferably post-dosed.

Full neutralisation of the acid precursor is only effected in step (ii) if the pumpable feedstock comprises, in addition to the acid precursor of the anionic, an organic liquid component such as a liquid nonionic surfactant or organic solvent. Partial neutralisation is possible whether or not the feedstock contains such other organic liquid component but it is especially useful when aluminosilicate builder is dosed into the high speed mixer/densifier in step (iii). Full neutralisation in step (ii) is applicable to cases of dosing phosphate or aluminosilicate builders in step (iii)

To obtain a high density powder, the powder from step (iii) should be further treated in a moderate speed mixer. To maximise the efficiency of this densification process, the material should be brought or maintained in its deformable state.

The acid precursor may for example be the precursor of a linear alkylbenzenesulphonate surfactant.

Preferably, the neutralisation in step (iii) is performed using a strong alkaline material such as an alkali metal hydroxide, e.g. NaOH. Preferably, any final complete neutralisation in step (iii) is performed using a relatively

weak alkaline material such as an alkali metal carbonate, e.g. Na_2CO_3.

For partial neutralisation, if has been found that the best results are obtained if the degree of pre-neutralisation in step (ii) is from 10 to 50 mole% of the acid precursor, most preferably from 20 to 40 mole%, especially from 25 to 35 mole%.

The residence time in the high-speed mixer/densifier during step (iii) is preferably about from 5 to 30 seconds. Moreover, the residence time in the moderate-speed mixer/densifier (if used) is preferably about from 1 to 10 minutes. The process is preferably performed as a continuous process.

Granular detergent compositions according to the present invention may be in the form of complete products ready for sale to the consumer. Alternatively, they may be formulated as base powders to which other ingredients are post-dosed. In any event such compositions preferably have a bulk density of 550 g/l, more preferably at least 650 g/l.

Preferably, the total amount of neutralised anionic in the final composition is from 2% to 50% by weight, especially from 5% to 15%.

Detergent compositions of the invention may contain, in addition to the anionic surfactant dosed in step (i) one or more other detergent-active compounds (surfactants) which may be chosen from soap and non-soap anionic cationic, nonionic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof. These may be dosed at any appropriate stage before or during steps (i)-(iii) or post-dosed after step (iv). Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch. The preferred detergent-active compounds that can be used

are soaps and synthetic non-soap anionic and nonionic compounds.

Other suitable anionic surfactants are well-known to those skilled in the art. Examples include primary and secondary alkyl sulphates, particularly C12-C15 primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

Suitable nonionic surfactants include the primary and secondary alcohol ethoxylates, especially the C_8-C_{20} aliphatic alcohols ethoxylated with an average of from 1 to 20 moles ethylene oxide per mole of alcohol, and more especially the C_{10}-C_{15} primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

Compositions according to the present invention may also contain, in addition to the detergent-active compounds, a detergency builder and optionally bleaching components and other active ingredients to enhance performance and properties. These may also be dosed at an appropriate time during steps (i)-(iv) or post-dosed.

The total amount of all surfactant present in the detergent granules is suitably from 20 to 70 wt% of the base powder granule although amounts outside this range may be employed as desired.

The detergent compositions of the invention generally also contain a detergency builder. The total amount of detergency builder in the compositions is suitably from 10 to 80 wt%, preferably from 15 to 60 wt%. The builder may be present in an adjunct with other components or, if desired, separate builder particles containing one or more builder materials may be employed.

Inorganic builders that may be present include sodium carbonate, if desired in combination with a crystallisation seed for calcium carbonate as disclosed in GB-A-1 437 950. Thus sodium carbonate will clearly need to be in excess of any used to neutralise the anionic acid precursor.

Other suitable builders include crystalline and amorphous aluminosilicates, for example zeolites as disclosed in GB-A-1 473 201; amorphous aluminosilicates as disclosed in GB-A-1 473 202; and mixed crystalline/amorphous aluminosilicates as disclosed in GB 1 470 250; and layered silicates as disclosed in EP-B-164 514. Inorganic phosphate builders, for example, sodium, orthophosphate, pyrophosphate and tripolyphosphate, may also be present, but on environmental grounds those are no longer preferred.

Aluminosilicates, whether used as layering agents and/or incorporated in the bulk of the particles may suitably be present in a total amount of from 10 to 60 wt% and preferably an amount of from 15 to 50 wt%. The zeolite used in most commercial particulate detergent compositions is zeolite A. Advantageously, however, maximum aluminium zeolite P (zeolite MAP) described and claimed in EP-A-384 070 may be used. Zeolite MAP is an alkali metal aluminosilicated of the P type having a silicon to aluminium ratio not exceeding 1.33; preferably not exceeding 1.15; and more preferably not exceeding 1.07.

Organic builders that may be present include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphonates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, aminopolycarboxylates such as nitrilotriacetates (NTA), ethylenediaminetetraacetate (EDTA) and iminodiacetates,

alkyl- and alkenylmalonates and succinates; and sulphonated fatty acid salts. A copolymer of maleic acid, acrylic acid and vinyl acetate is especially preferred as it is biodegradable and thus environmentally desirable. This list is not intended to be exhaustive.

Especially preferred organic builders are citrates, suitably used in amounts of from 5 to 30 wt%, preferably from 10 to 25 wt%; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, preferably from 1 to 10 wt%. Citrates can also be used at lower levels (eg 0.1 to 5%) by weight for other purposes. The builder is preferably present in alkali metal salt, especially sodium salt, form.

Suitably the builder system comprises a crystalline layered silicate, for example, SKS-6 ex Hoechst, a zeolite, for example, zeolite A and optionally an alkali metal citrate.

Detergent compositions according to the invention may also contain a bleach system, desirably a peroxy bleach compound, for example, an inorganic persalt or organic peroxyacid, capable of yielding hydrogen peroxide in aqueous solution. The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. An especially preferred bleach system comprises a peroxy bleach compound (preferably sodium percarbonate or perborate optionally together with a bleach activator), and a transition metal bleach catalyst as described and claimed in EP 458 397A and EP-A-509 787.

Powder flow may be improved by the incorporation of a small amount of an additional powder structurant, for example, a fatty acid (or fatty acid soap), a sugar, an acrylate or acrylate/maleate polymer, or sodium silicate which is suitably present in an amount of from 1 to 5 wt%.

The materials that may be present in detergent compositions of the invention include sodium silicate; corrosion inhibitors including silicates; antiredeposition agents such as cellulosic polymers; fluorescers; inorganic salts such as sodium sulphate, lather control agents or lather boosters as appropriate; proteolytic and lipolytic enzymes; dyes; coloured speckles; foam controllers; and fabric softening compounds. This list is not intended to be exhaustive.

In the step (iii) of the process, solid components of the feedstock are very thoroughly mixed with the liquid components by means of a high-speed mixer/densifier. Such a mixer provides a high energy stirring input and achieves thorough mixing in a very short time.

As high-speed mixer/densifier we advantageously used the Lödige (Trade Mark) CB 30 Recycler. This apparatus essentially consists of a large, static hollow cylinder having a diameter of about 30 cm which is horizontally placed. In the middle, it has a rotating shaft with several different types of blades mounted thereon. It can be rotated at speeds between 300 and 2500 rpm, dependent on the degree of densification and the particle size desired. The blades on the shaft provide a thorough mixing action of the solids and the liquids which may be admixed at this stage. The mean residence time is somewhat dependent on the rotational speed of the shaft, the position of the blades and the weir at the exit opening.

Other types of high-speed mixers/densifiers having a comparable effect on detergent powders can also be contemplated. For instance, a Shugi (Trade Mark) Granulator or a Drais (Trade Mark) K-TTP 80 may be used.

In step (iii), the components of the feedstock are thoroughly mixed in a high-speed mixer/densifier for a relatively short time of about 5-30 seconds, preferably under conditions whereby the starting material is brought

into, or maintained in, a deformable state, to be defined hereafter.

If after step (iii) the detergent material still possesses a considerable porosity, instead of choosing a longer residence time in the high-speed mixer/densifier to obtain a further bulk density increase, the process of the present invention may comprise a second processing step in which the detergent material is treated for e.g. 1-10 minutes, preferably for 2-5 minutes, in a moderate-speed granulator/densifier. During this second processing step, the conditions are such that the powder is brought into, or maintained in, a deformable state. As a consequence, the particle porosity will be further reduced. The main differences with the first step reside in the lower mixing speed and the longer residence time of 1-10 minutes, and the necessity for the powder to be deformable. A high-speed mixer densifier machine, followed by a separate moderate-speed granulator/densifier machine could be used for this purpose. Alternatively, it is possible to use a single machine operated at two speeds, first at high speed for mixing/densification and then at moderate speed for granulation/densification. Suitable machines include mixers of the Fukae® FS-G series; Diosna® V series ex Dierks & Sohne, Germany; Pharma Matrix® ex T.K. Fielder Ltd, England; Fuji® VG-C series ex Fuji Sangyo Co., Japan; the Roto® ex Zanchetta & Co. srl, Italy and the Schugi® Flexomix granulator.

The optional step of using a moderate speed mixer/densifier can be successfully carried out in a Lödige (Trade Mark) KM 300 mixer, also referred to as Lödige Ploughshare. This apparatus essentially consists of a hollow static cylinder having a rotating shaft in the middle. On this shaft various plough-shaped blades are mounted. It can be rotated at a speed of 40-160 rpm. Optionally, one or more high-speed cutters can be used to

prevent excessive agglomeration. Another suitable machine for this step is, for example the Drais (Trade Mark) K-T 160.

For use, handling and storage, the densified detergent powder must obviously no longer be in a deformable state. Therefore, in step (iv) the densified powder is dried and/or cooled. This step can be carried out in a known manner, for instance in a fluid bed apparatus (drying, cooling) or in an airlift (cooling). It is advantageous if the powder needs a cooling step only, because the required equipment is relatively simple and more economical.

The invention will now be explained in more detail by way of the following non-limiting examples.

EXAMPLES

Example 1

Feed to high speed mixer

FEEDSTOCK

LAS-acid		14.24
Nonionic premix		13.53
Nonionic 7EO	7.67	
Nonionic 3EO	4.11	
Fatty acid	1.75	
Zeolite A24		47.30
Sodium Carbonate		14.36
Sokalan CP-5 solution		3.97
SCMC		0.93
Water/NaOH		1.05

Feed to moderate speed mixer

Zeolite A24	4.62
	100.00

The LAS-acid of the feedstock was 30% neutralised with the water/NaOH and fed to a high-speed mixer/densifier (recycler) with the remaining feedstock components, where it was completely neutralised with the sodium carbonate. The resultant material was then fed to a moderate-speed granulator/densifier (ploughshare) which was also dosed with the Zeolite builder prior to drying and cooling of the final detergent granules.

The powder exhibited excellent physical properties. After spray-on of perfume onto the finished powder no dark speckles developed.

In the light of this disclosure, modifications of the described examples, as well as other examples, all within the scope of the present invention as defined by the appended claims will now become apparent to persons skilled in the art.

CLAIMS:

1. A process for preparation of a granular detergent composition which process comprises the steps of

(i) producing a pumpable feedstock comprising an acid precursor of an anionic surfactant;

(ii) partially neutralising the acid precursor in the feedstock;

(iii) feeding the feedstock into a high-speed mixer/densifier and further neutralising the partially neutralised acid precursor therein, to form a granular detergent material; and

(iv) drying and/or cooling the resultant granular detergent material.

2. A process according to claim 1, wherein the granular detergent material produced in step (iii) is processed in a moderate speed mixer/densifier prior to step (iv).

3. A process according to any preceding claim wherein an aluminosilicate builder is dosed into the high-speed mixer/densifier during step (iii) and the acid precursor is only partially neutralised during step (ii).

4. A process according to any preceding claim, wherein the acid precursor is a precursor of a linear alkylbenzenesulphonate surfactant.

5. A process according to any preceding claim, wherein the neutralisation in step (ii) is performed using a strong alkaline material.

6. A process according to claim 5, wherein the strong alkaline material comprises an alkali metal hydroxide.

7. A process according to any preceding claim, wherein any neutralisation in step (iii) is performed using a weak alkaline material.

8. A process according to claim 7, wherein the weak alkaline material comprises an alkali metal carbonate.

9. A process according to any preceding claim, wherein the degree of neutralisation in step (ii) is from 10 to 60 mole% of the total acid precursor.

10. A process according to any preceding claim, wherein the granular detergent composition so prepared comprises a perfume.

11. A granular detergent composition prepared by a process according to any preceding claim.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D11/00 C11D11/04 C11D17/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 93 23520 A (HENKEL KGAA ;WILMS ELMAR (DE)) 25 November 1993 see page 5, line 14 - page 7, line 21; claims 1-16; examples 1-3	1,4-9,11
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[X] Further documents are listed in the continuation of box C

[X] Patent family members are listed in annex

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